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(54) PRODUCTION OF POROUS BODY OF ACTIVATED CARBON

(57)Abstract:

PROBLEM TO BE SOLVED: To readily and economically produce a porous body of an activated carbon excellent in adsorption properties and handling abilities and having a large specific surface area at high yield by heating a foamed body obtained by using a novolak type phenol resin as a main raw material, in an inert gas to carbonize, and activating the obtained carbonized product.

SOLUTION: A novolak type phenol resin obtained by reacting phenols with aldehydes in the presence of an acidic catalyst, with a prescribed amount of hexamethylenetetramine is mixed with a foaming agent, and the mixed product is heat-treated in a prescribed framework at 140-200°C to be sufficiently foamed and hardened. The obtained novolak type phenol resin foamed product in a prescribed shape, optionally in a shape of a reinforced composite product molded by packing the foamed product in a cell of a honeycomb core, is carbonized and activated. The carbonizing and activating steps are carried out, for example, by heating the foamed body in an inert gas flow at about 800°C or more, preferably about 850-1,000°C, flowing a oxidative gas such as a water steam and air on the heated foamed body, and cooling the product in the inert gas.

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CLAIMS

[Claim(s)]

[Claim 1] The manufacture method of the activated carbon porous body obtained carbonization and by activation-izing in a phenol resin foam.

[Claim 2] The manufacture method according to claim 1 that a phenol resin foam is a foam obtained considering novolak type phenol resin as a main raw material.

[Claim 3] The manufacture method according to claim 1 that a phenol resin foam is the honeycomb composite molding object with which it fills up with the phenol resin foam in the cell of a honeycomb core.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Industrial Application] As a field for which the activated carbon porous body obtained by this invention is used, fields, such as the clean water and water treatment for which coconut husks activated carbon and the activated carbon fiber are used conventionally, a gas adsorption, and a solvent recovery, are raised.

[Description of the Prior Art] The activated carbon fiber obtained considering the fiber conventionally manufactured as an activated carbon material from the powdered active carbon obtained as a raw material, a granular active carbon, a pitch, a polyacrylonitrile, and phenol resin in powder coal, charcoal, wood, fruits husks (coconut husks, walnut husks), etc. as a raw material is raised. These are obtained by the chemical or the well-known activation method which used a steam, combustion gas, etc.

[Description of the Prior Art] Although the powder and the granular active carbon which are manufactured from powder coal, charcoal, wood, fruits husks (coconut husks, walnut husks), etc. could be cheaply manufactured from the low price of the raw material, they had the problem that only 600-1000m²/g and a low thing were obtained for specific surface area. On the other hand, in order to obtain the fiber whose activated carbon fiber which uses a pitch, a polyacrylonitrile, and phenol resin as a raw material is the precursor of an activated carbon fiber although what has a specific surface area highly efficient [to the 2/g-th 2500m place] is obtained, manufacture of a raw material and the process of spinning are needed, and, in the case of a pitch and a polyacrylonitrile, the process of non-deliquest processing and many is further needed. Therefore, the product had the problem that it could not but become expensive.

[Problem(s) to be Solved by the Invention] this invention is obtained as a result of examining obtaining easily and economically the activated carbon porous body excellent in adsorptivity ability and handling nature paying attention to the trouble shown above. That is, by using a novolak type phenol resin foam as a start raw material of an activated carbon porous body, it has a predetermined configuration, finds out that the activated carbon porous body of high specific surface area excellent in handling nature is easily obtained by high yield, and results in this invention. Furthermore, this invention is conventionally applicable also to the composite molding object which combined heat insulation, the novolak type phenol resin foam currently used as a heat-resisting material, and various core material for reinforcement, such as a honeycomb core, a roll core, and a corrugated core, in a commercial scene. Furthermore, it is also possible to use as the disposal method of the edge material of this composite molding object and a tail, and it is significant also from a viewpoint of a deployment of resources.

[Means for Solving the Problem] If a novolak type phenol resin foam is used as a start raw material for obtaining activated carbon material as this invention shows, diffusion of the activation gas inside a foam is possible at the time of that the omission of the cracked gas at the time of carbonization is easy, and activation, and in order to act effectively [can take the large touch area of activation gas, and], the activated carbon porous body of the high specific surface area in high yield will be obtained. Furthermore, because of the structure of a foam, it is possible to absorb well the stress by the contraction at the time of carbonization, there are few curvatures and cracks and it is possible to obtain an activated carbon porous body in the form which reduced the configuration in front of carbonization activation as it was. That is, the thing of the various configurations which are needed

in case an activated carbon porous body is used will be obtained easily. this invention is explained in detail below. It is obtained by making phenols and aldehydes react under existence of an acid catalyst the passage that the novolak type phenol resin used for this invention is well-known. a phenol, cresol, a xylenol, a resorcinol, a catechol, etc. are used and independent [in these] as phenols, -- or even if two or more sorts are mixed, it can use formaldehyde, a paraformaldehyde, an acetaldehyde, etc. are used and independent [in these] as aldehydes, -- or even if two or more sorts are mixed, it can use As an acid catalyst, inorganic acids, such as organic acids, such as oxalic acid, Para toluenesulfonic acid, and a citric acid, and a hydrochloric acid, a sulfuric acid, are used. The thing of the type which the foaming agent used for this invention causes decomposition by heating of PARATORU en sulfonylhydrazide, AZOJI carvone amide, dinitrosopentamethylenetetramine, p, and p'-oxybis benzene sulfonylhydrazide, a PARATORUEN sulfonyl acetone hydrazone, etc., and generates gas is used. Moreover, since the state of the bubble at the time of foaming is prepared if needed, the method of mixing the surfactant in novolak type phenol resin beforehand as a foam stabilizer can also be taken. As a method of obtaining a foam, novolak type phenol resin, a hexamethylenetetramine, and a foaming agent are covered over a grinder at a predetermined rate, and it considers as powder of about 50 micrometers of mean particle diameters. Moreover, each component is uniformly mixed at this time. In this way, a novolak type phenol resin foam is obtained by sprinkling the obtained novolak type phenol resin constituent uniformly in a predetermined mold, and heat-treating among the oven kept at 140-200 degrees C, until sufficient foaming and hardening are obtained by the heat press etc. What is necessary is next, just to cool by changing to inert gas again in inert gas aeration like nitrogen or argon gas, as a conventional method, as a method of obtaining an activated carbon porous body from this novolak type phenol resin foam, after heating in temperature of 850-1000 degrees C preferably and performing temperature of at least 800 degrees C or more, and activation by oxidizing gases, such as a steam, a carbon dioxide, combustion gas, and air. Control of specific surface area is possible by adjusting the temperature at this time, oxidizing gas aeration speed, and quantity of airflow. In others, as the simple carbonization activation method, a sample is covered with non-oxides, such as corks, fine coal, and chaff, and the method of heating under the environment where a moderate quantity of a oxidizing gas exists is. Moreover, the method of heating under the environment where a moderate quantity of a oxidizing gas exists can also be taken by using as a furnace the half-airtight container which prepared only the exhaust pipe for exhausting the gas which occurred by the pyrolysis of a foam.

[Example] An example is shown below. this invention is not limited to the following example.

The novolak-resin constituent was obtained by a grinder's grinding the letter novolak type phenol resin of block 100 weight section with a softening temperature of 88 degrees C obtained by reacting a [example 1] phenol and formalin under existence of an oxalic acid catalyst, the hexamethylenetetramine 10 weight section, and the foaming agent AZOJI carvone amide 7 weight section, and mixing each component uniformly. The grain size at this time was 10-70 micrometers. Next, 55g of this novolak-resin constituent was uniformly sprinkled in the 20x10x5cm mold, and the foam was obtained by heat-treating for 4 minutes with the molding press with a hot-platen temperature of 160 degrees C. The density of the obtained foam was 0.05 g/cm³. This foam was put into the electric tubular furnace, and the steam was introduced into the tubular furnace after the temperature up to 900 degrees C by the basis of nitrogen aeration, 5 degrees C of programming rates, and min, and it cooled, carrying out aeration only of the nitrogen gas, after holding for 30 minutes at 900 degrees C, and the activated carbon porous body was obtained.

The foam obtained by the method of the [example 2] example 1 was put into the electric tubular furnace, and the steam was introduced into the tubular furnace after the temperature up to 900 degrees C by 5 degrees C of programming rates, and min also as nitrogen aeration, and it cooled, carrying out aeration only of the nitrogen gas, after holding for 50 minutes at 900 degrees C, and the activated carbon porous body was obtained.

The foam obtained by the method of the [example 3] example 1 was put in into the magnetic container, the surroundings were covered with corks, in the muffle furnace, by 5 degrees C of programming rates, and min, after holding for 40 minutes after a temperature up to 900 degrees C, cooling was performed and the activated carbon porous body was obtained.

The grinder ground the novolak type phenol resin 100 weight section and the

hexamethylenetetramine 10 weight section which were used in the [example 1 of comparison] example 1, and the novolak-resin constituent was obtained by mixing each component uniformly. next, this novolak-resin constituent -- 10x10x0.5cm metal mold -- inside -- 65g -- putting in -- the molding press with a hot-platen temperature of 160 degrees C -- gradually -- pressurization -- on the way -- the phenol resin board which density 1.27 g/cm³ hardened was obtained by repeating deflation This phenol resin board was put into the electric tubular furnace, carbonization activation was performed on the same conditions as an example 1, and the activated carbon board was obtained.

The [example 2 of comparison] coconut husks (mean particle diameter : 3.6mm) were put into the electric tubular furnace, and the steam was introduced into the tubular furnace after the temperature up to 900 degrees C by the basis of nitrogen aeration, 5 degrees C of programming rates, and min, and it cooled, carrying out aeration only of the nitrogen gas, after holding for 30 minutes at 900 degrees C, and activated carbon was obtained. The specific surface area of the activated carbon material obtained by the example 1 mentioned above or 3 and the example 1 of comparison, or 2 was shown in Table -1 about the result measured with Micromeritics fluid type specific-surface-area automatical measurement equipment, yield, and appearance.

[Table 1]

	炭化収率 (%)	比表面積 (m ² /g)	外観
実施例 1	40.6	1032	反り、割れ無し
実施例 2	33.2	1910	反り、割れ無し
実施例 3	43.3	925	反り、割れ無し
比較例 1	45.1	580	反り、割れ有り
比較例 2	19.6	760	粒状

[Effect of the Invention] According to this invention, the activated carbon porous body of high specific surface area can be easily obtained by high yield so that clearly from the result of an example.

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